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RESEARCH ARTICLE

Comparative Biosorption Of Acid Red YG Dye From Aqueous Solution And Textile Industry Wastewater Using *Syzygium cumini* Seeds And *Borassus flabellifer* Seeds Peels

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Abstract

Application of *Syzygium cumini* seeds and *BorassusFlabellifer* Seeds Peels as biosorbents for removal of Acid Red YG dye from aqueous solution and textile industry wastewater were investigated. Batch experiments were carried out to determine the effect of pH, particle size, biosorbent dose, contact time and initial dye concentration and even characterization like FTIR and SEM along with desorption studies were also carried out. Maximum dye biosorption was achieved at pH 2.0 for *Syzyguim cumini* seeds (92.60 %) whereas biosorption for *Borossus flabellifer* seeds peels (86.04 %) was achieved at pH 3.0, particle size 212µm for both, biosorbent dose of 0.8 g/L for *Syzygium cumini* seeds and 1g/L for *Borassus flabellifer* seeds peels, contact time remained the same as 60 min and initial concentration of 50mg/L. The pseudo-second order and equilibrium biosorption data fitted well to Langmuir than Freundlich isotherm models for both the biosorbents. The biosorption results in this study indicated that the seeds of *Syzygium cumini* and seeds peels of *Borossus flabellifer* are attractive alternatives for removing acidic dyes from wastewater

INTRODUCTION

Dyes are toxic and trace materials found in wastewater. Due to their complex and aromatic nature, they are considered to be difficult materials for the biodegrading process (Ansari et al., 2011). Dyeing industry effluents are one of the most problematic wastewaters to be treated, not only for their high chemical oxygen demand, but also for other factors. These factors are high biological oxygen demand, suspended solids, turbidity, toxic constituents and also for colour, which is the first contaminant discernible by the human eye. Dyes may affect the photosynthetic activity in aquatic life due to reduced light penetration and may also be toxic to some aquatic life due to the presence of aromatics, metals, etc. in them (Clarke and Anliker 1980; Zollinger 1987; Mishra and Tripathy 1993; Banat et al; 1996; Fu and Viraraghvan 2001; Robinson et al; 2001). The adverse effect of these dyes has not only been seen on the environment but also it poses a major concern when it comes to human health. They have polluted the nature by slowly increasing the biomagnifications of the chemicals resulting in imbalance of hormones and immunity loss that may result in cancer (Lanone et al., 2002; Yu et al., 2002;Topaç et al., 2009)

To reduce the toxicity of dyes many alternative methods were obtained such as chemical coagulation using alum, lime, ferric sulphate (Low et al., 1994),oxidation methods using chlorine and ozone (Churchley., 1994) biological treatments, floatation(Weber ., 1978) and many more which have drawbacks in terma of expense, inefficiency and sludge disposal problem(Ahalya et al., 2002). Therefore the industries interest and the main approach is towards low cost and effective method which can remove the dyes from the effluent or wastewater and support in maintaining the balance of nature. Hence the approach of processes like adsorption or biosorption using agricultural or low cost material is been favourable by the industries. Recently, biosorption has been recommended as cheaper and more effective technique for dye contaminated wastewater treatment (Srinivasan et al., 2010).

The present study is the comparative investigation of biosorption of Acid Red YG (ARYG) dye from aqueous solution and textile industry wastewater using Syzygiumcumini seeds (SCS) and borossusflabelliferseeds peels (BFSP). The effect of pH, particle size, biosorbent dose, contact time and initial dye concentrations on biosorption were studied in detail. Characterizations of the biosorbents were carried out by FTIR and SEM analysis. Kinetic modeling, Langmuir and Freundlich isotherm models were used to fit the equilibrium data.

MATERIAL AND METHODS

2.1 Structure of Acid RedYG

The Acid Red YG (ARYG) dye was obtained from Zese India LTD, Bangalore, Karnataka. Its IUPAC name is Disodium 4,4'-bis(2-hydroxynaphthalen-1-ylazo)biphenyl-2,6'-disulphonate. The molecular formula is $C_{32}H_{20}N_4Na_2O_8S_2$, molecular weight is 693.63 [g/mol] and structure of the ARYG is shown in figure 1

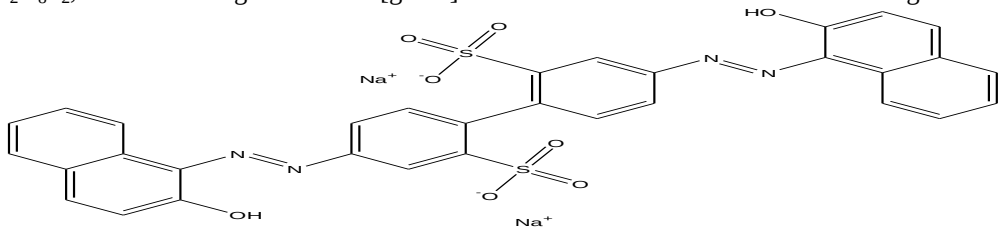


Fig 1- Structure of ARYG

2.2 Dye solution preparation

An accurately weighed quantity of ARYG dye was dissolved in double distilled water to prepare stock solution (1000mg/L) and further diluted to obtain working solutions of lower concentration 50 mg/L which was determined using the absorbance values before and after the biosorption using UV-Visible Spectrometer(HitachiUV-2910). The pH adjustment of solution was carried out using 0.01N HCl and 0.01N NaOH

2.3 Preparation of biosorbent:

This study was focused in investigating biosorption of ARYG using *Syzyguim cumini* seeds (SCS) and *Borassusflabellifer* seeds peel (BFSP). Seeds and seeds peels were collected washed thoroughly and dried it in the sunlight. Subsequently the seeds peels were grounded and sieved to get fine sizes (212µm, 250µm, 355µm, 500µm) according to the standards of ASTM (American Society Testing Materials). And sieved biosorbent was washed thoroughly and constantly several times using double distilled water to remove anyunwanted particles, and then dried well in the sunlight. The cool biosorbentwas stored in a dry place for experimental use.

2.4 Characterization of biosorbent

The FTIR and SEM analyses were carried out by (Shimadzu-IRAffinity-1) FTIR Spectrophotometer and ZEISS, (EVO/LS-15).

2.5 Batch experiments

Batch experiments were carried out at room temperature. Different parameters such as effect of pH ranging from 2.0 to 10.0, particle size ranging from 212µm to 500µm, biosorbent dose ranging from 0.2 g/L to 2.0 g/L, contact time ranging from 10 to 120 min and initial dye concentration ranging from 10 to 50 mg/L were studied. pH adjustment of the solutions were carried out using 0.01N HCl and 0.01N NaOH. Mixtures were taken in 250 mL. Erlenmeyer flasks and shaken at 170rpm using mechanical shaker (KEMI KRS 110, India) for a fixed period of time. The mixture was then filtered from ASB and determined spectrophotometrically by recording the absorbance changes at maximum biosorption (520nm) using UV-Vis Spectrometer (HitachiUV-2910). All determinations that were done in triplicate average values were considered.Percentage biosorption was calculated by the formula

$$\%\text{Biosorption} = \left(\frac{C_i - C_f}{C_i}\right) \times 100 \qquad (1)$$

where C_i and C_f are the initial and final OR concentration (mg/L) respectively

2.6 Desorption Studies

Desorption experiments were carried out by taking loaded ARYG biosorbents in Erlenmeyer flasks containing 50 mL of 1M HCl. The mixtures were shaken at 170 rpm on a mechanical shaker until equilibrium was reached. After filtration, supernatant solution were analysed for ARYG concentration using UV-Vis spectrophotometer in order to determine desorption percentage. The extent of desorption was calculated using the following equation;

$$\%\text{Desorption} = \frac{C_d}{C_b} \times 100 \qquad (2)$$

Where, C_d is the concentration of dye desorbed from biosorbent (mg/L) and C_b is the concentration of dye biosorbed on biosorbent (mg/L)

Before applying to the subsequent biosorbent loading cycle, the biosorbents were separated from the solutions and were washed with doubled distilled water several times. The same biosorbents were used for all the three cycles to determined biosorption and desorption process

2.7Physico-chemical characteristics of textile industry wastewater

The wastewater was collected from textile industry located in Bangalore, Karnataka

India. The physico-chemical characteristics were determined using standard method of APHA (Cleseri et al., 2012). The results are presented in Table 1.

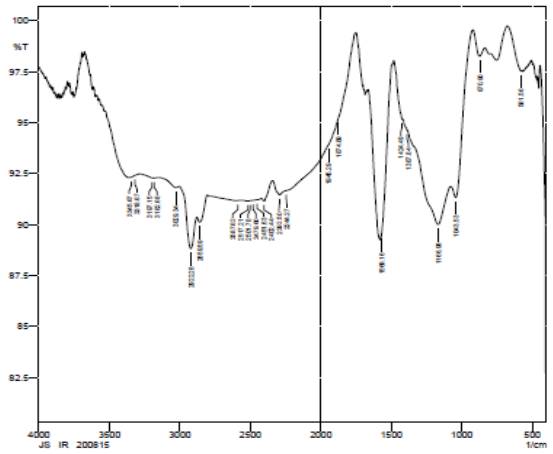
RESULTS AND DISCUSSION

TABLE 1: Physico-Chemical Characteristics Of Textile Industry Wastewater

Characteristics	Contents(mg/L)
Colour	Reddish pink
pH	7.84
EC(µs/cm)	1506
TDS	689
Total hardness	571.4
Calcium	167.8
Magnesium	83.1
Chloride	394.2
Sulphate	278.5
DO	0.09
BOD	805.3
COD	1067.9

3.1 FTIR Analysis

Figure 2a and 2b shows FTIR spectra of ARYG before and after biosorption on SCS and Figure 2c and 2d shows ARYG before and after biosorption on BFSP. These figures show that some peaks are shifted or disappeared and some new peaks are also detected. The changes in the spectrum indicate the functional groups of sulphur and carboxylic acid and their derivatives. The peak ranging from 544cm^{-1} is due to S-S bonds in figure 2a which are shown to be reduced in figure 2b for SCS where as figure 2c S-S bonds are stretched in figure 2d due to biosorption of ARYG on SCS and BFSP. The peaks between 1539 and above 3740 cm^{-1} indicate the N-H bonds and C-N bonds of amine along with overlapped C-H stretched bands. The peaks which are reduced in figure2b indicate strong stretching vibration of carboxylic acids groups of amide. An FTIR spectrum of fig 2b shows peaks in low frequency regions and absence of peaks ranging from 514cm^{-1} to 720cm^{-1}because there is a biosorption of ARYG on SCS surface, similar effects are also observed on BFSP. Biosorption showed that carbon, hydrogen and atom bonds played major role. Decreased intensity of sharp peaks concluded that ARYG has been functionalized by SCS and BFSP.



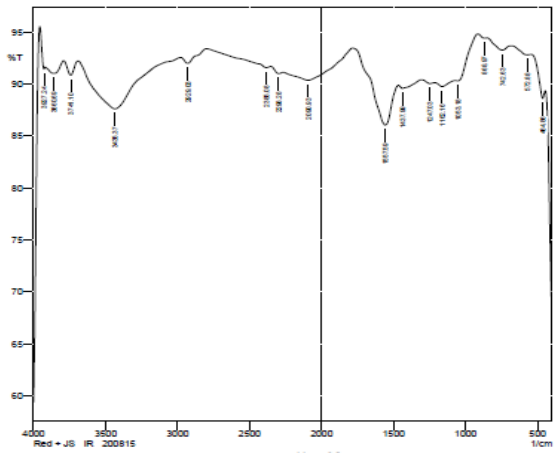
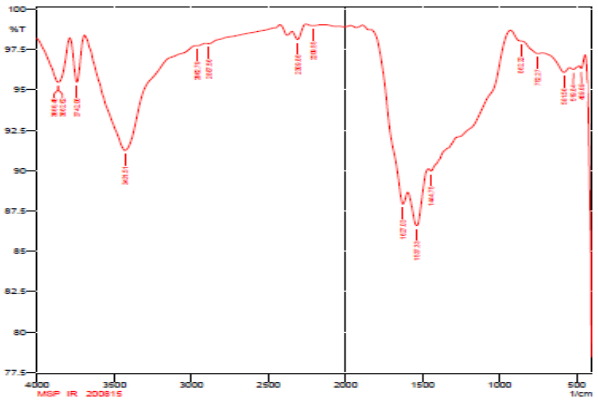


Fig-2 a. FTIR of SCS before biosorption **b. FTIR of SCS after biosorption**



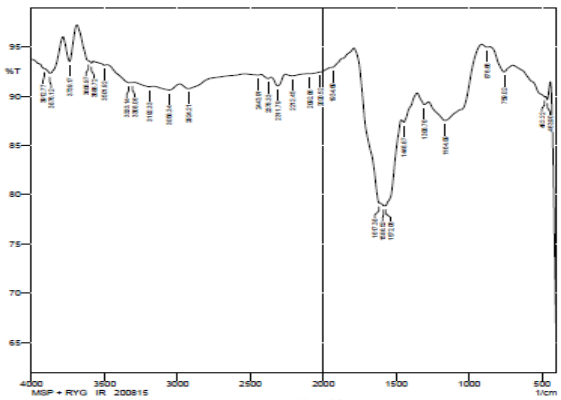
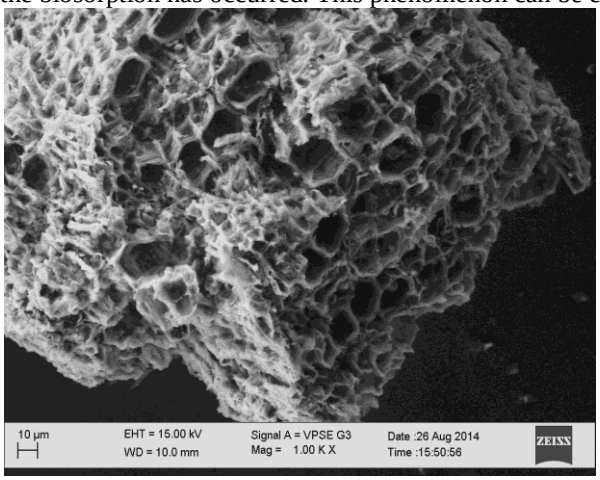


Fig-2 c. FTIR of BFSP before biosorption **d. FTIR of BFSP after biosorption**

3.2 Scanning Electronic Microscopic Studies (SEM)

The SCS and BFSP morphology were characterized using SEM and the microphotographs are shown in Figure 3a, 3b, 3c and 3d. Figure 3a shows that pores within SCS are highly heterogeneous before biosorption of ARYG, whereas figure 3c describes thick lining of veins for BFSP. However it is not the case after biosorption, since a significant change is observed in the structure of the seeds as observed in figure 3b. They appear rougher with the formation of crater-like pores and figure 3d shows vertical opening appearing scale like which completely describes the biosorption has occurred. This phenomenon can be explained by the package of pores involved in dye molecules.



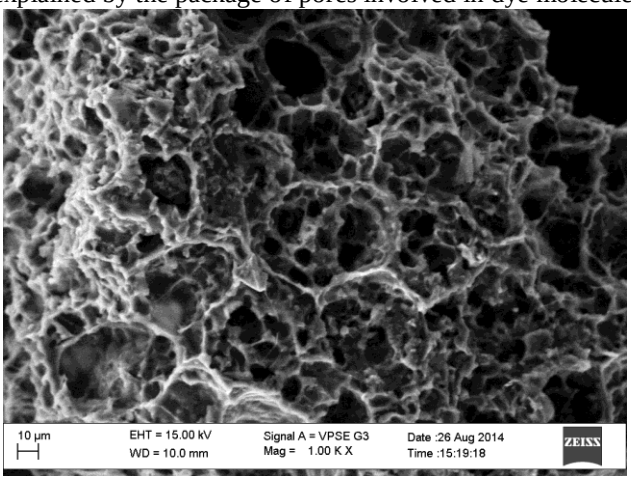


Fig 3 a: SEM of SCS before biosorption **b: SEM of SCS after biosortion**

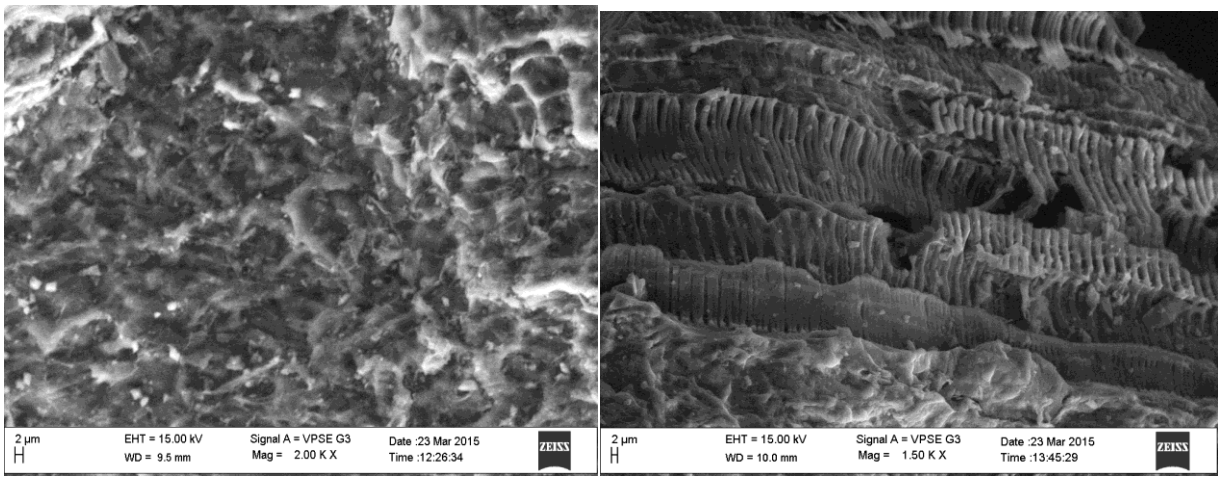


Fig 3 c: SEM of BFSP before biosorption **d: SEM of BFSP after biosortion**

3.3 Effect of pH

The pH of the ARYG has a great influence on the biosorption process on SCS and BFSP as it has an effect on the surface charges, degree of ionization, structural stability or dissociation of the dye molecules on SCS and BFSP (Ahalya et al 2002; Monash et al., 2010). Figure 4 shows the effect of pH on the removal of ARYG. The highest dye removal efficiency was observed at pH 2.0 (92.60 %) for SCS and pH 3.0 (86.04%) for BFSP. The removal percentage then decreased as pH increased from 3.0 to 10.0 for SCS and 4 to 10.0 for BFSP. This is due to the H_3O^+ ions which were replaced by OH^- ions as the pH increases; it makes the surface negatively charged. Similar results were recorded in Acid Blue 92 (Abhiti et al., 2010),Indosol Yellow BG(Sadaf et al., 2014), acid blue 129 (Nekouei et al., 2015) in the process of biosorption.

3.4 Effect of biosorbent particle size

The effects of particle size on the biosorption of the ARYG on to SCS and BFSP are shown in the figure 5.The results describe that the percentage removal of the ARYG increases with decrease in SCS and BFSP particle size which were (92.60 % and 86.04 %) achieved at 212µm. This clearly states that decrease in the particle size would increase in the surface area, and consequently an increase in ARYG biosorption onto the SCS and BFSP surface occurs. Various factors are responsible for the low biosorption capacity of the ARYG on the biosorbent's large particles. Similar kind of results were observed in fruit peel waste (Wanna et al., 2009), unripe grape juice waste (Ansari et al., 2011)and solid agricultural waste (Shabuddin et al., 2011).

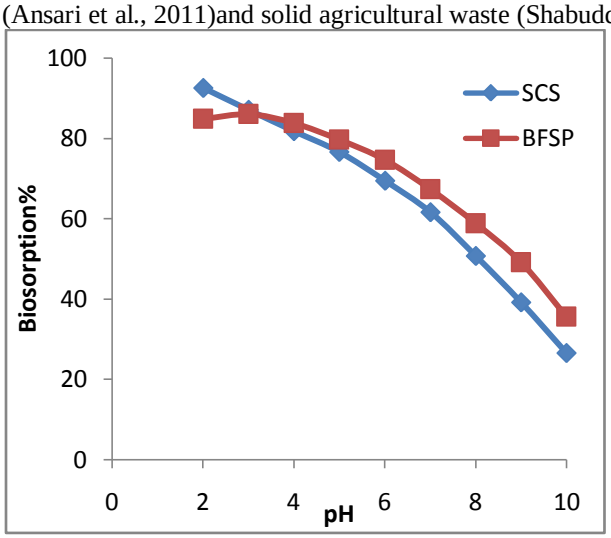


Fig 4: Effect of pH on ARYG biosorption on SCS and BFSP

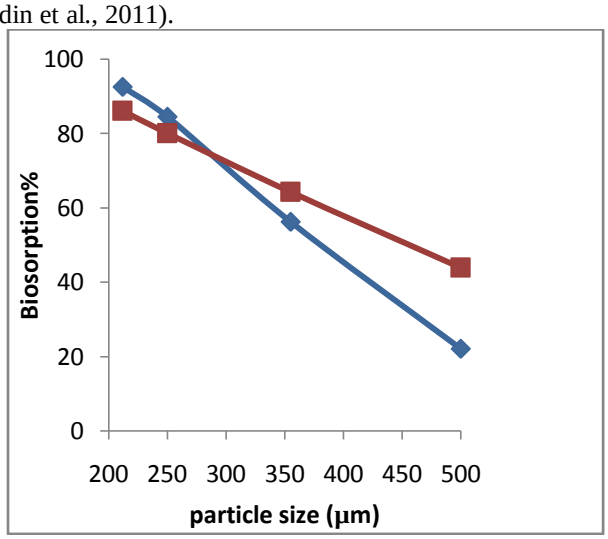


Fig 5: Effect of particle size on ARYG biosorption on SCS and BFSP

3.5. Effect of biosorbentdose

The biosrbent doses on ARYG by SCS and BFSP are shown in figure 6. The results describe that ARYGbiosorption by SCS and BFSP increases with increase in the biosorbent dose from 0.2 to 2g/L, which were achived at 0.8 g/L for SCS (71.01 to 92.60%) and 1 g/L for BFSP (61.63 to 86.04 %).This is due to the increase in surface area which in turn increases the availability of the exchangeable sites on the SCS and BFSP for the biosorption of ARYG (Tunc et al., 2009). Further increase in biosorbent dose beyond 0.8 g/L for SCS and 1g/L for BFSP keeps ARYGbiosorption static which suggest that it has attained equilibrium between the liquid and solid phase. Similar results were observed in Reactive Yellow Dye (Veeramalini et al; 2012), Indosol Yellow BG (Sadaf et al., 2014) and acid blue 129 (Nekouei et al., 2015).

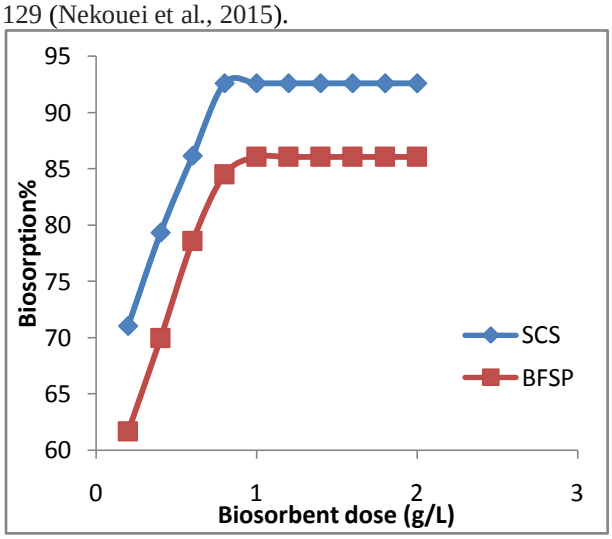


Fig 6: Effect of biosorbent dose on ARYG biosorption on SCS and BFSP

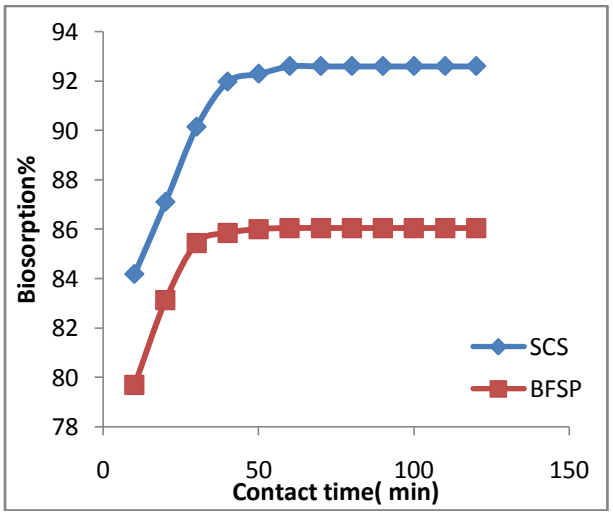


Fig 7: Effect of contact time for ARYG biosorption on SCS and BFSP

3.6 Effect of Contact time

The variation of contact time of ARYG by SCS and BFSP are shown in figure 7. It was clear that maximum biosorption by SCS was 92.60% and BFSP was 86.04 % for ARYG was achieved at 60 min. A rapid biosorption of the ARYG by SCS and BFSP took place at 30min and then the biosorption was slow till it reached the equilibrium. The reason being that at initial stage there was a more vacant biosorption site which gets occupied, later resulting in the slow biosorption, and once the biosorption reaches the equilibrium the contact time and percentage of biosorption becomes constant. Similar results were pragmatic in forestry waste (Berraksu et al., 2012), almond shell residues (Deniz., 2013) and coal fly ash (Ferrero ., 2015)

3.7 Kinetic isotherms

The Lagergren's pseudo-first-order model and Ho's pseudo-second-order model (Reddy et al., 2012) have been widely used to predict adsorption kinetics. The pseudo-first order equation is generally applicable to the initial stage of the adsorption processes whereas the pseudo-second order equation predicts the behavior over the whole range of adsorption. These two models were used to fit the experimental data of this study whose equations are described in equation (3) and (4)

$$\log(q_e - q_t) = \log q_e - \left(\frac{K_1}{2.303}\right) t \quad (3)$$

$$\frac{t}{qt} = \left(\frac{1}{K_2 q_e^2}\right) + \left(\frac{1}{q_e}\right) t \quad (4)$$

where k_1 (min^{-1})is the rate constant of pseudo-first- order adsorption and k_2(g mg^{-1} min^{-1}) is the rate constant of pseudo –second-order adsorption. q_e, amount of dye biosorbed on biosorbent at equilibrium.

Plots of pseudo-first-order and second order are shown in Figure 8 and 9.Results show that pseudo-first-order rate constant (K_1) for ARYG biosorption by SCS was $2.30X10^{-2}$and BFSP was $1.85X10^{-2}$ min^{-1} but q_{ecal} values obtained largely different than that of q_{eexp} values which indicated the non-applicability of pseudo- first order model, whereas higher pseudo-second order rate constant (K_2) of 9.10 X 10^{-2} g/mg $min^{-1}$was achieved by SCS and 3.54 X 10^{-2} g/mg min^{-1} was achieved by BFSP, which show rapid ARYG biosorption by SCS and BFSP. The q_{ecal} values of pseudo-second order are closer to q_{eexp} values and higher regression coefficient (R^2) value was obtained reveal better fitting of equilibrium data to pseudo second order model. This confirmed that involvement of chemical biosorption through

exchange or sharing of electrons between biosorbent and biosorbate. (Kumar et al., 2010; Kaur et al., 2015). Table 2 describes the pseudo-first order and pseudo-second order.

Table -2 Pseudo First Order And Pseudo Second Order Constants Chosen For Biosorption By SCS and BFSP

Biosorbent	Pseudo first order model				Pseudo second order model		
	q_{eexp} (mg/g)	$K_1(min^{-1})$	q_{ecal} (mg/g)	R^2	$K_2(g/mg\ min^{-1})$	q_{ecal} (mg/g)	R^2
SCS	37.87	2.30×10^{-2}	36.93	0.967	6.25×10^{-2}	37.60	0.999
BFSP	23.02	1.85×10^{-2}	20.17	0.989	3.54×10^{-2}	23.00	0.999

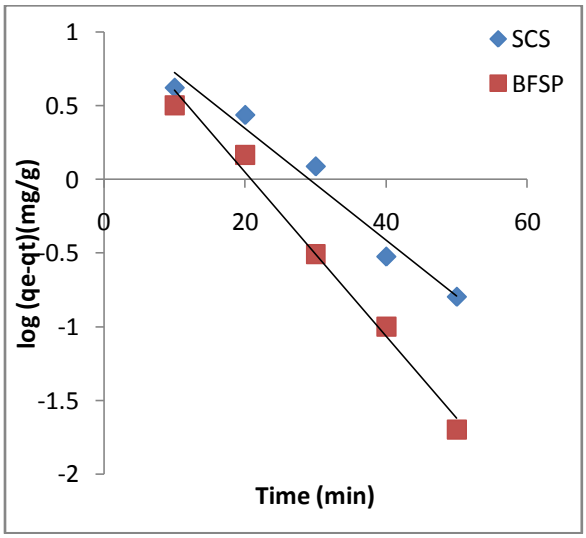


Fig 8:Pseudo-first order for ARYG by SCS and BFSP

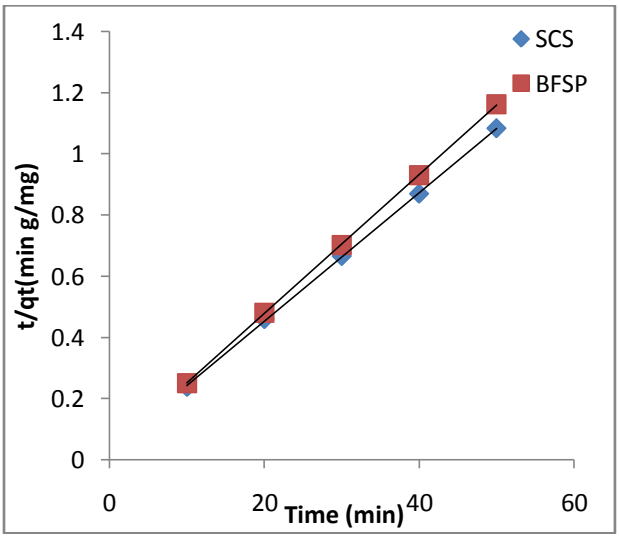


Fig 9: Pseudo-second order for ARYG by SCS and BFSP

3.6 Effect of initial dye concentration

Figure 8 shows the initial concentration of ARYG by SCS and BFSP which describes that the percentage of biosorption decreases (99.20% to 92.60%) for SCS and (99.60 to 86.04 %) for BFSP with increase in the concentration. This is due to the fact that in dye ion concentration causes saturation of biosorption sites on SCS and BFSP which blocks further ARYG biosorbents on biosorption sites, as a result the biosorption efficiency decreases. Similar results were observed in rice husk and rice husk ash (Sharma et al., 2010) and nanoparticles of mango seed kernel (Salwa et al., 2015).

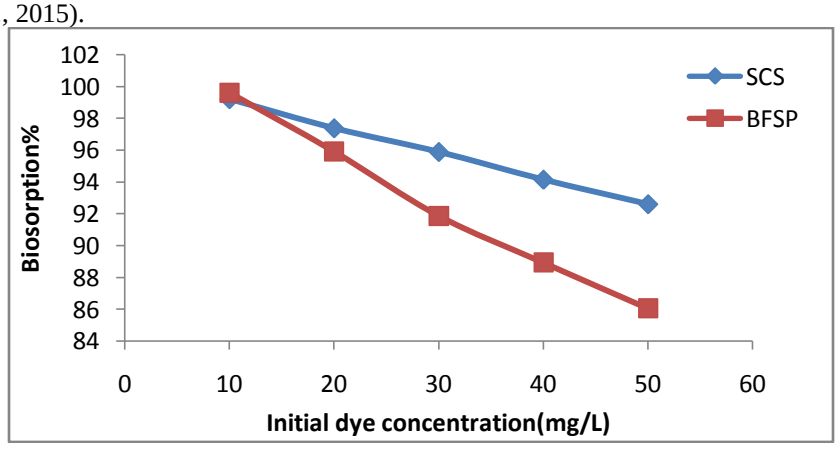


Fig 10: Effect of initial dye concentration ARYG biosorption on SCS and BFSP

3.7 Langmuir and Freundlich isotherm

The biosorption data were fitted to two isotherms namely Langmuir and Freundlich. The Langmuir (Langmuir, 1918) and Freundlich isotherm (Freundlich, 1906) model equations are described in equation (4) and (5) respectively.

$$\frac{C_e}{q_e} = \frac{1}{(bq_{max})} + \frac{C_e}{q_{max}} \quad (4)$$

$$q_e = K_f C_e^{\frac{1}{n}} \quad (5)$$

Where, $C_e$is the equilibrium concentration (mg/L), q_e and q_{max} are the equilibrium and maximum biosorption capacity (mg/g), respectively and b is the Langmuir constant and K_F and n are the Freundlich constant.

Figure 11 shows the linear plot obtained by plotting C_e/q_e versus C_e. The Langmuir model effectively describes the biosorption data R^2 value of 0.999 are given in Table 3. This behaviour indicates a monolayer biosorption. The value of b (100.00 L/g for SCS and 74.61 L/g for BFSP) and Q_{max} (43.00 mg/g for SCS and 32.98 mg/g for BFSP) indicates maximum interaction and greater affinity of ARYG with SCS and BFSP (Sohn et al., 2005).Figure 10 shows the linear plot obtained by plotting log q_e versus log C_e. The Freundlich model parameters like R^2 (0.988 for SCS and 0.983 for BFSP) values, K_f (9.99 (mg/g) (mg/L) $^{1/n}$ for SCS and 6.30 (mg/g) (mg/L) $^{1/n}$ for BFSP) and n (0.95 for SCS and 0.87 for BFSP) are given in Table 3. The results indicate that biosorption data for ARYG fits well to Langmuir model than Fruendlich model for both the biosorbents. Hence biosorption was monolayer coverage of ARYG on SCS and BFSP. (Martín et al., 2013; Sassi ., 2014).

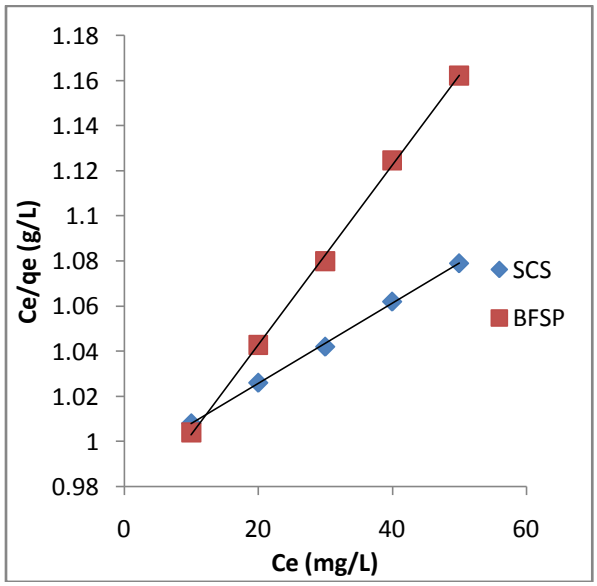


Fig 9: Langmuir isotherm plots for ARYG by SCS and BFSP

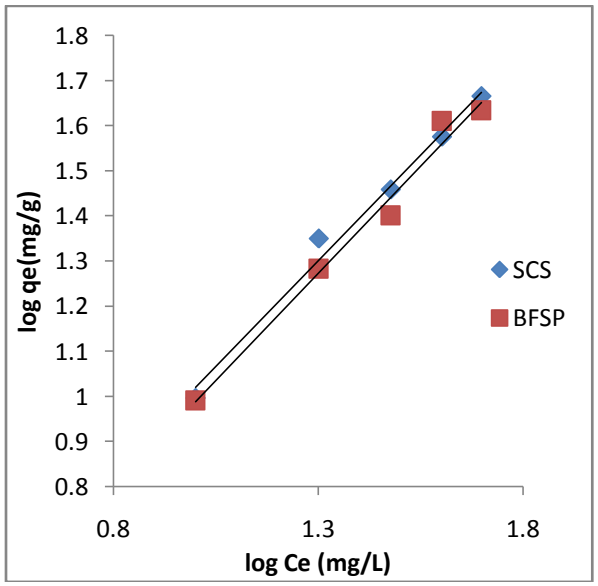


Fig 10: Freundlich isotherm plots for ARYG by SCS and BFSP

Table -3 Langmuir Isotherms AndFreundlich Isotherm Parameters For Biosorption By SCS and BFSP

Biosorbent	Langmuir Isotherm			Fruendlich Isotherm		
	Q_{max} (mg/g)	b(L/g)	R^2	K_f(mg/g)(mg/L)$^{1/n}$	n	R^2
SCS	43.00	100.00	0.999	9.99	0.95	0.988
BFSP	32.98	74.61	0.999	6.30	0.87	0.983

3.8 Desorption studies

Figure 11a and b showed that at 60 min, SCS showed maximum desorption efficiency (92.60 %) and BPSF showed (86.04 %) for first cycle. But for third cycle ARYG desorption efficiency by SCS and BFSP decreased to 34.25 % and 0.0%. Similarly, increasing the number of cycles from one to three decreased ARYG biosorption efficiency from 77.16 5 to 0.0 % for regenerated SCS, and 62.10 % to 0.0 % for regenerated BFSP. These decreases is attributed to lesser or remove number of biosorption sites available on the surface of SCS and BFSP. Similar results were observed for algal biomass (Gajare et al., 2012)

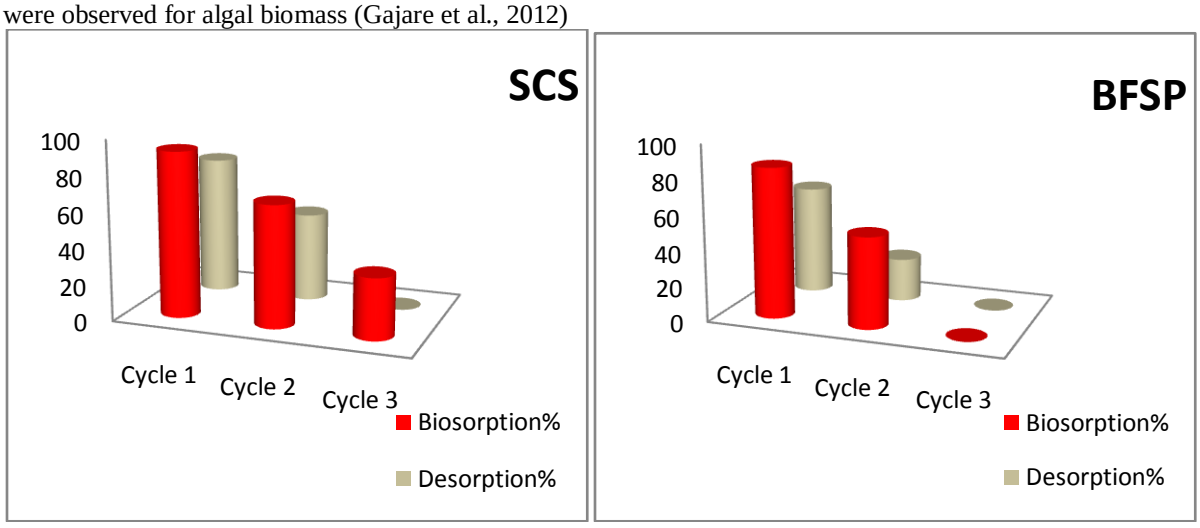


Fig 11 a: Bisorption/desorption efficiency for SCS **b: Bisorption/desorption efficiency for BFSP**

3.8 Application of leather industry wastewater

The efficiency of SCS and BFSP for ARYG from textile industry wastewater was determined. The experiments were performed in an Erlenmeyer flask containing 50 mL of wastewater samples. To this 212µm of 0.8 g/L of SCS and 1g/L of BFSP were added and pH were adjusted to 2.0 and 3.0 using 0.01N HCl and 0.01N NaOH. After 60min of agitation, the wastewater samples were filtered and the resultant supernatant solutions were analysed by UV.Visible.spectrophotometer (HitachiUV-2910). The result described that ARYG biosorption from textile industry wastewater was 90.24 % for SCS, and 78.16 % for BFSP which was less than the aqueous solution. This is due to competition between ions of dyes and co-ions interference for biosorption sites on SCS and BFSP (Mishra et al., 1993).

CONCLUSION

The present study investigated the comparative biosorption of ARYG by SCS and BFSP from aqueous media and textile industry wastewater.Characterizations were carried out by FTIR and SEM. The biosorption equilibrium was described by the isotherm model. Single-stage batch biosorption systems were designed for the ARYG removal which was also outlined based on the isotherm model. Pseudo-second order model and Langmuir model data fits well than Fruendlich model, proving biosorption was monolayer coverage of ARYG on SCS and BFSP. Desorption/ biosorption study also reveals the usage of SCS and BFSP. And when the SCS and BFSP were applied for textile industry wastewater, percentage of biosorption was less in comparison to aqueous solution, as the competition between ions of ARYG and co ions interaction of other dyes. It was concluded that the SCS and BFSP could be used as promising alternative options for ARYG removal from aqueous solutions and textile industry wastewater

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